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Indian Standard

METHODS OF TEST FOR SULPHUR

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MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

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Indian Standard
METHODS OF TEST FOR SULPHUR

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Indian Standard

METHODS OF TEST FOR SULPHUR

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 14 September 1972, after the draft finalized by the Inorganic Chemicals (Miscellaneous) Sectional Committee had been approved by the Chemical Division Council.

0.2 There is practically no production of sulphur in this country and the entire demands are met by imports only. This standard has been prepared to provide acceptable methods for analysis of sulphur intended for general industrial use.

0.3 Although commercial mine-run sulphur is a comparatively pure product, always having more than 99 percent sulphur (dry basis), the trade recognizes several chemical impurities and physical contaminants. Among these, the major ones are as follows:

a) *Ash*

b) *Moisture*

c) *Arsenic, selenium and tellurium*

d) *Acid* — Which is extracted from sulphur by an aqueous solution of isopropyl alcohol and titrated with sodium hydroxide solution using phenolphthalein as indicator.

e) *Hydrocarbon materials of varying degrees of unsaturation* — These may be partly in chemical combination with some of the sulphur and partly physically mixed with the sulphur. These impurities are determined either by an empirical method based upon converting them to a residual carbon-sulphur complex or by direct combustion and are then reported as carbon.

0.4 There is no generally accepted specification either as to colour or as to carbon which distinguishes a dark-sulphur from a bright one. If necessary, the colour of sulphur is determined by an empirical technique based upon the reflectance of a sample of powdered material. Alternatively, a solution of the sulphur in carbon disulphide having a specified concentration, say 5 percent, is compared with a similar solution containing the acceptable quality of sulphur.

0.5 Generally, in reporting the results of analysis the various impurities and contaminants are reported individually as determined and the sulphur is reported by difference. However, in this standard two assaying methods have been prescribed for the estimation of elemental sulphur.

0.6 For the guidance of the analyst, the values for the assay and impurities are given below:

Characteristic	*Percent by mass*
Assay (*dry basis*)	99
Colour	Bright whitish yellow
Moisture	Moisture in sulphur occurs only in bulk solid sulphur, usually because of rain or other atmospheric contamination
Ash	0·25
Sulphuric acid	0·03
Chlorides	0·000 1 to 0·001
Selenium	0·000 2
Tellurium	0·000 2
Arsenic	0·000 025 (0·25 parts per million)
Carbon	0·1 to 1·0

0.7 In the preparation of this standard, assistance has been derived from 'Analysis of Sulphur', Texas Gulf Sulphur Co, USA and 'The Sulphur Data Book' Freeport Sulphur Co, USA. The method for determination of sulphur is based on the method prescribed in the Technical Association of Pulp and Paper Industry, USA.

0.8 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off it shall be done in accordance with IS : 2-1960*.

1. SCOPE

1.1 This standard prescribes the methods of test for sulphur intended for general industrial use. It covers the methods of determination of sulphur, moisture, ash, acidity, chlorides, selenium, tellurium, arsenic carbon and fineness of sulphur.

1.1.1 This standard does not include the material specification.

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*Rules for rounding off numerical values (*revised*).

2. QUALITY OF REAGENTS

2.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1960*) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which effect the results of analysis.

3. TEST METHODS

3.1 Determination of Elemental Sulphur (Uncombined Sulphur)

3.1.0 Two methods are prescribed, namely, Method I and Method II. For routine work, Method I is recommended. For very accurate determinations, Method II is recommended which may be adopted in case of dispute.

3.1.1 *Method I*

3.1.1.1 *Reagent* — Carbon disulphide.

3.1.1.2 *Procedure* — Weigh accurately 1 g of the sample, as dried in **3.2**, ground to pass 1·70-mm IS Sieve, in the fritted glass extraction thimble and extract in the extraction apparatus for at least 15 minutes with carbon disulphide at the rate of one cycle per minute. Remove the container, dry and weigh. Report the percentage insoluble residue as foreign matter (this includes ash). Report the percentage of sulphur as the difference between 100 and the percentage of foreign matter.

NOTE — Organic impurities may dissolve in carbon disulphide causing slight inaccuracy.

3.1.1.3 *Calculation*

$$\text{Sulphur, percent by mass} = 100 - \left[\frac{M_1}{M} \times 100\right]$$

where

M_1 = mass in g of the carbon disulphide insoluble residue, and

M = mass in g of the material taken for the test.

3.1.2 *Method II*

3.1.2.1 *Reagents*

a) *Bromine water* — saturated.

b) *Concentrated nitric acid* — *see* IS : 264-1968†.

c) *Sodium chloride* — solid.

---

*Specification for water, distilled quality (*revised*).

†Specification for nitric acid (*first revision*) (Since revised).

d) *Concentrated hydrochloric acid* — *see* IS : 265-1962*.

e) *Dilute hydrochloric acid* — 1 : 1 (*v/v*).

f) *Barium chloride solution* — 10 percent (*m/v*).

3.1.2.2 *Procedure* — Weigh accurately about 0·5 g of the sample as dried in **3.2**, and ground to pass 1·70-mm IS Sieve, into a 500-ml covered beaker; warm to 30°C and add about 6 ml of bromine water. Keep the mixture at this temperature for about ten minutes, then add 15 ml of concentrated nitric acid previously brought to 30°C. After the violent reaction has subsided heat the mixture, cautiously at first, then boil to drive off bromine and nitric acid. Add about 2·5 g of sodium chloride to avoid loss of sulphuric acid and evaporate the mixture to a small volume. Repeat the evaporation three or four times, adding after each evaporation about 5 ml of concentrated hydrochloric acid. Evaporate to dryness on the steam-bath and heat the residue gently. Take up this residue with 5 ml of dilute hydrochloric acid and about 10 ml of water, filter out the insoluble matter through Whatman filter paper No. 42 or equivalent and wash thoroughly on the filter paper with hot water. (This may be ignited and weighed, if the amount of insoluble matter is desired to be determined).

3.1.2.3 Transfer the filtrate to a 500-ml volumetric flask, dilute to the mark and thoroughly mix. Pipette a 100-ml aliquot into a beaker, heat to boiling and add 100 ml of barium chloride solution drop by drop to the boiling solution with constant stirring. Stir the solution thoroughly, preferably let it stand overnight, and filter through an ignited and tared Gooch crucible with asbestos mat. Wash with boiling water till free from chlorides; dry, ignite at 700 to 750°C and weigh as barium sulphate.

3.1.2.4 *Calculation*

$$\text{Sulphur, percent by mass} = 100 \times \frac{0{\cdot}137\,5\,M_1}{M}$$

where

M_1 = mass in g of the ignited barium sulphate precipitate, and

M = mass in g of the material taken for the test.

3.2 Determination of Moisture

3.2.1 *Procedure* — Weigh accurately about 50 g of the sample into a previously dried, cooled, and tared 100 × 15 mm petri dish. Place it in an oven at 105 ± 1°C for 1 hour. Then remove, cool in a desiccator and weigh till constant mass is obtained.

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*Specification for hydrochloric acid (*revised*).

3.2.2 *Calculation*

$$\text{Moisture, percent by mass} = 100 \times \frac{M - M_1}{M}$$

where

M = mass in g of the material taken for the test, and

M_1 = mass in g of the material after heating.

NOTE 1 — Avoid prolonged drying or overheating, since vapourization of the sulphur will cause the moisture estimate to be too high. It is advisable to place the dish in the top position of the oven on two small pieces of insulating material, such as 5 mm asbestos board, so that there will be an air space between the bottom of the dish and the shelf of the oven, to avoid local overheating.

NOTE 2 — The moisture determination may be carried out at 80°C for overnight (16 hours), if more convenient.

NOTE 3 — Some sulphurs of high hydrocarbon content may contain an appreciable amount of hydrocarbon which is volatile at the 105°C which is the temperature used to determine moisture. The determination of carbon in sulphur as described in **3.7** is made on an undried sample which avoids loss of this volatile carbon. The error introduced by dividing by

$$0{\cdot}1 - \frac{\text{percent moisture}}{100}$$

to convert to the dry basis is negligible since the volatile hydrocarbon is usually only a few tenths of one percent.

NOTE 4 — The amount of hydrocarbon volatilized in the moisture determination may be estimated by analyzing the dried as well as the undried sample for carbon. The difference obtained in the two carbon contents is the carbon lost by volatilization, and when multiplied by the factor 1·17, is the hydrocarbon (CH_2) volatilized. This loss of CH_2 may be subtracted from the value obtained in the moisture determination when sulphur of high carbon content is analyzed, if a very accurate determination of moisture content is necessary.

NOTE 5 — The dried sample after moisture determination is used for the determination of ash, elemental sulphur, arsenic, selenium and tellurium. Since approximately 100 g of the dried sulphur will be required for all of the determinations, the moisture determination should be made in duplicate.

3.3 Determination of Ash

3.3.1 *Procedure* — Weigh accurately 25 g of the sample as dried in **3.2**, into a previously ignited, cooled, and tared silica dish of diameter approximately 100 mm and place on a hot-plate. Control the hot plate so that the sulphur burns slowly but completely. After the sulphur has burnt off completely, ignite in a muffle furnace at about 800°C. Cool in a desiccator, and weigh. Repeat the ignition until the mass is constant. (One 30-minutes ignition is usually sufficient.)

3.3.2 *Calculation*

$$\text{Ash, percent by mass} = 100 \times \frac{M_1}{M}$$

where

M_1 = mass in g of the residue obtained, and

M = mass in g of the material taken for the test.

3.4 Determination of Acidity

3.4.1 *Reagents*

3.4.1.1 *Isopropyl alcohol* — neutral.

3.4.1.2 *Standard sodium hydroxide solution* — 0·025 N.

3.4.1.3 *Phenolphthalein indicator solution* — Dissolve 0·1 g of phenolphthalein in 100 ml of rectified spirit (*see* IS : 323-1959)*.

3.4.2 *Procedure* — Weigh accurately about 20 g of the undried sample into a 250-ml conical flask. Add 25 ml of isopropyl alcohol and shake until the sulphur is completely wetted by the alcohol. Add 50 ml of water, shake for 1 to 2 minutes more, and allow to stand for 20 minutes with occasional shaking. Titrate with standard sodium hydroxide solution, using phenolphthalein indicator, until a slight pink colouration is obtained. Similarly titrate a mixture of 25 ml of isopropyl alcohol and 50 ml of water as a blank.

3.4.3 *Calculation*

$$\text{Acidity (as } H_2SO_4\text{), percent by mass} = \frac{(V_1 - V_2) \times N \times 4{\cdot}904}{M}$$

where

V_1 = volume in ml of the sodium hydroxide solution required for titration of the material,

V_2 = volume in ml of the sodium hydroxide solution required in the blank,

N = normality of standard sodium hydroxide solution, and

M = mass in g of the material taken for the test.

NOTE — Acid is not determined on samples from molten sulphur storage or molten sulphur shipments.

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*Specification for rectified spirit (*revised*).

3.5 Determination of Chlorides

3.5.1 *Reagents*

3.5.1.1 *Calcium Carbonate* — solid.

3.5.1.2 *Isopropyl alcohol*

3.5.1.3 *Potassium chromate indicator* — saturated solution.

3.5.1.4 *Standard silver nitrate solution* — 0·02 N, freshly standardized.

3.5.2 *Procedure* — Weigh 100 g of the undried sample into a 500-ml stoppered conical flask. Add 1 g of calcium carbonate and exactly 200 ml of a mixture of 1 volume of isopropyl alcohol and 7 volumes of water. Stopper the flask and shake vigorously and continuously for 10 minutes.

3.5.2.1 Allow the sulphur and calcium carbonate to settle and decant the supernatant solution through a dry filter paper and funnel, into a 250-ml beaker. Pipette 100 ml of the filtrate into a porcelain dish. Add 5 drops of the potassium chromate indicator and titrate with standard silver nitrate solution until the first permanent colour change occurs. Carry out a blank test on all the reagents.

3.5.3 *Calculation*

$$\text{Chlorides (as Cl), percent by mass} = \frac{7{\cdot}1\,(V_1 - V_2) \times N}{W}$$

where

V_1 = volume in ml of standard silver nitrate solution required for titration of the material,

V_2 = volume in ml of standard silver nitrate solution required in the blank titration,

N = normality of standard silver nitrate solution, and

W = mass in g of the material taken for the test.

NOTE — It is important that the solution should be neutral or very slightly alkaline. Acidity tends to prevent the formation of silver chromate which is the indicator. Also if, as in rare cases, the sulphur should yield hydrogen sulphide to the solution, it should be removed by making the solution acidic to methyl orange and boiling off the hydrogen sulphide. Neutralization can then be performed on the cooled solution by using sufficient sodium bicarbonate.

3.6 Determination of Selenium, Tellurium and Arsenic

3.6.0 *General* — Arsenic, selenium and tellurium can be run on the same sulphur sample. The first step in the determination is the oxidation of the sample to sulphuric acid which is then used for the determination colorimetrically.

3.6.1 *Apparatus*

3.6.1.1 *Nessler Cylinders* — 100 ml capacity.

3.6.1.2 *Concentrated nitric acid* — *see* IS : 264-1968*.

3.6.1.3 *Concentrated sulphuric acid* — *see* IS : 266-1961†.

3.6.1.4 *Standard selenium solution* — Dissolve 1 g of pure selenium in concentrated nitric acid. Evaporate to dryness. Add water and repeat evaporation two or three times to ensure complete removal of nitric acid. Dilute to 1 litre with water; 1 ml of this solution is equivalent to 1 mg of selenium (Se). Dilute 10 ml of this solution to 1 litre with water. One millilitre of the diluted solution is equivalent to 0·01 mg of selenium (Se).

3.6.1.5 *Standard tellurium solution* — Dissolve 1 g of pure tellurium in concentrated nitric acid. Add 50 ml of concentrated sulphuric acid and evaporate to fumes. Dilute in a 1-litre volumetric flask with water. Dilute 10 ml of this solution to exactly 1 litre with water. One millilitre of this solution is equivalent to 0·01 mg of tellurium (Te).

3.6.1.6 *Concentrated bromine-carbon tetrachloride mixture* (2 : 3) — Mix 2 volumes of bromine with 3 volumes of carbon tetrachloride.

3.6.1.7 *Hydrazine sulphate* — solid.

3.6.1.8 *Stannous chloride solution* — 80 g of stannous chloride ($SnCl_2.2H_2O$) dissolved in 100 ml of water to which is added 5 ml of concentrated hydrochloric acid. The dissolution is slow.

3.6.2 *Procedure* — Weigh 50 g of the sample as dried in **3.2** into a conical flask with a 50/42 ground glass joint. It is desirable to run another 50 g sample simultaneously to which 0·25 parts per million arsenic; 2 parts per million selenium, and 2 parts per million tellurium have been added to ensure that none of these components is lost in the oxidation. Under a hood add 180 ml of bromine-carbon tetrachloride mixture to the sample and immediately cover with a combined condenser and dropping funnel. Fill the dropping funnel with concentrated nitric acid and add rapidly 8 to 10 drops of the nitric acid and gently rotate flask to ensure mixing. Keep the outlet of the condenser pointed away from the face while doing this because bromine may be evolved rather vigorously.

Set the flask on an asbestos mat on a hot plate. For the initial oxidation use only one element of the three-element hot plate. Set the

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*Specification for nitric acid (*first revision*) (Since revised).

†Specification for sulphuric acid (*revised*).

dropper to deliver one or two drops of the nitric acid per second. Continue the dropping of acid until no further reaction is noted in the flask. The dropping funnel may have to be refilled two or three times.

After the oxidation is complete, remove the condenser and boil off the bromine. Continue boiling until sulphur trioxide fumes are evolved. Cool and add 100 ml of water, boil and reduce to fumes again. Repeat the addition of water and reduction to fumes. The fuming is necessary to ensure removal of all the nitric acid.

NOTE 1 — After the first fuming, samples containing organic impurities may be brown. In this case, it is necessary to add a small amount of bromine mixture and concentrated nitric acid followed by fuming to remove all the colour. For increasing amounts of organic impurities, it is necessary to repeat this step until the final solution is water white.

NOTE 2 — It is well to keep a bottle of alcohol handy to inhale if bromine is breathed during the process. A cloth soaked with ammonium hydroxide placed in the front of the hood will tend to prevent bromine from escaping into the room.

3.6.2.1 *Determination of selenium* — The final solution from the oxidation of 50 g of sulphur should produce about 150 g (80 ml) of sulphuric acid. Dilute this cooled solution with 80 ml of water and bring to boil on a hot-plate. While boiling, add 0·1 to 0·3 g of hydrazine sulphate. A pink colour indicates the presence of selenium.

Determine selenium by comparing the colour in a Nessler cylinder against standards containing standard sulphuric acid to which known amounts of standard selenium solution have been added diluting both the sample and the standard solutions to 100-ml mark. Before the determination, the solution shall be cooled to room temperature because the colour is much more pronounced in cooled solutions.

3.6.2.2 *Determination of tellurium* — If the solution which was tested for selenium shows no colour, the solution can be used as such for the determination of tellurium. However, if selenium is present, it should be removed first. The red selenium is colloidal. Convert it to black crystalline selenium by digesting overnight on a water bath, and then filtering off through a medium porosity sintered glass crucible.

Bring the solution to boil and add 0·5 ml of stannous chloride solution. A grey or black colour indicates the presence of tellurium.

Determine tellurium by comparing the colour in Nessler cylinder against standards containing standard sulphuric acid to which known amounts of standard tellurium solution have been added diluting both the sample and the standard solutions to 100-ml mark.

3.6.2.3 *Determination of arsenic* — Rinse the solution in which selenium and tellurium have been determined into a 500-ml volumetric flask and make up the volume up to mark with water. Use an aliquot of 100 ml

(representing 10 ml of the original sample) and determine arsenic by the method prescribed in IS : 2088-1962*.

3.7 Determination of Carbon (Combustion Method)

3.7.0 *General* — The method consists of completely burning the sulphur and carbon compounds in a current of pure oxygen to form sulphur dioxide and carbon dioxide. The sulphur dioxide in this gas mixture is then converted to sulphuric acid and absorbed by chromic acid solution. The gas mixture, now free of sulphur compounds, is dried with phosphorus pentoxide (P_2O_5) which does not absorb carbon dioxide. The carbon dioxide is finally absorbed from the gas mixture by ascarite in the absorption tube. The tube is weighed before and after conducting the combustion, and its gain in mass is a direct measure of the amount of carbon dioxide produced in the combustion, and thus of the amount of carbon burnt.

3.7.1 *Apparatus* — as shown in Fig. 1.

3.7.2 *Procedure*

3.7.2.1 All ground joints except those at the ends of the combustion tube shall be greased with a minimum of stop cock grease. Any excess grease which may be sequeezed into the interior where it would come into contact with flowing oxygen shall be removed. The reduced end of the combustion tube *B* shall be lubricated with concentrated sulphuric acid. The intake end of the combustion tube requires no lubricant. It is well to assemble the combustion train by starting at the vacuum end. The stoppers on all of the tubes following the combustion tube shall be closed during the assembly.

3.7.2.2 After completing the assembly, switch on the heavy duty furnace *5 D*. Start the oxygen flowing at a uniform rate which will be indicated by bubbles in jar No. 3. Start the vacuum pump and, after the vacuum is established as indicated by bubbles in the mercury trap No. 13, open the valves in the reverse order of flow. With pinch cock in the vacuum line, adjust the rate of flow to approximately 85-100 ml per minute. This rate will increase to 250-400 ml per minute during burning.

3.7.2.3 Volatilize any moisture or sulphuric acid in the combustion tube by moving a lighted Meker burner slowly along the tube ahead of the furnace. After the tube has cooled from the heating, test for leaks by removing the ground connection *5 A* from the combustion tube, and inserting a rubber stopper. After 5 minutes, equilibrium shall be obtained, and no flow shall be noted in the absorption tube No. 11.

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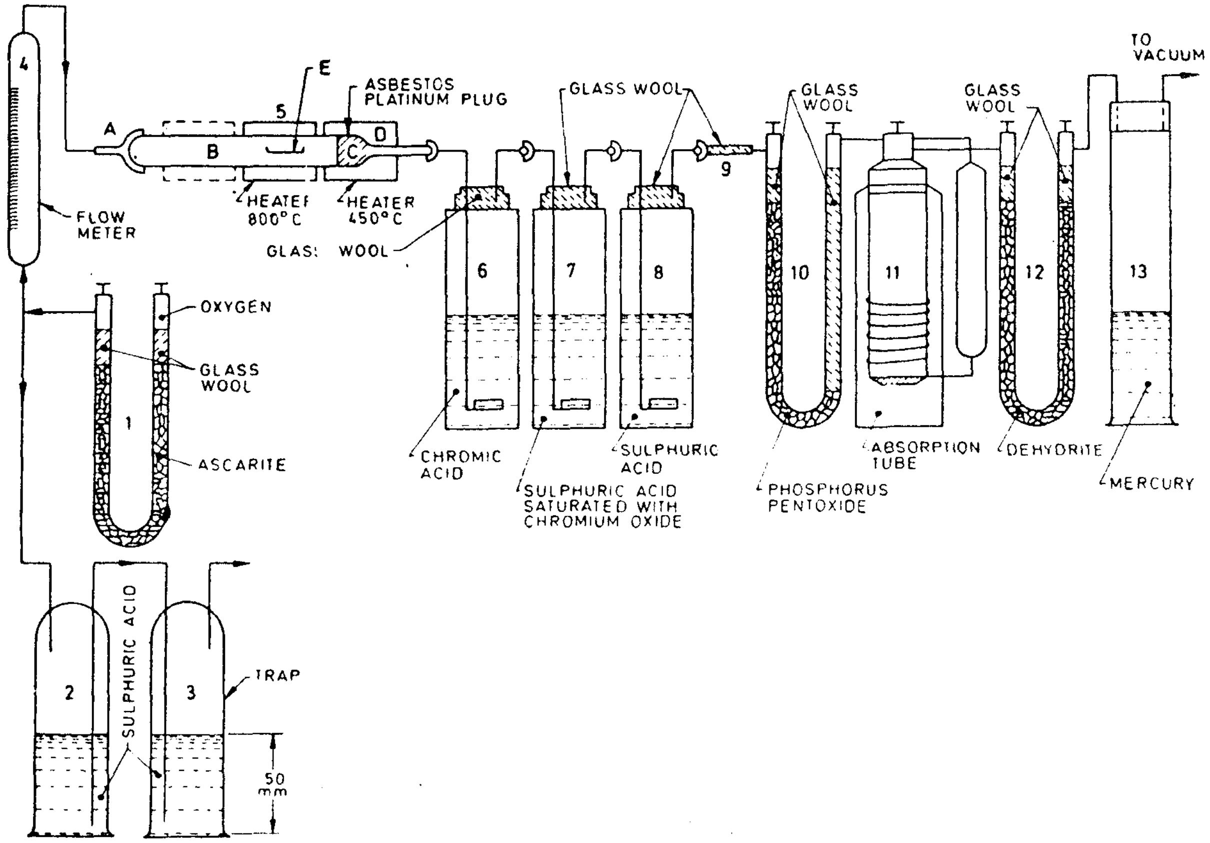
*Modified Gutziet method of test for arsenic.



1, 10, 12 — U-tubes containing ascarite, phosphorus pentoxide, and dehydrite, respectively, 150 mm in length each.

2, 3 — Purifying jars containing sulphuric acid for regulating oxygen pressure.

4 — Flowmeter for gas flow of 5 400 ml/minute with a steel ball float.

5 *A* — Ground connection preceding combustion tube is specially made from a 10-mm tubing, 75 mm long blown to a 35/25 socket joint.

B — Combustion tube of transparent quartz — 75 cm long 2·5 cm ID tubing reduced to 1 cm ID tubing about 12·5 cm long. The open 2·5 cm end has a 35/25 ball joint. The open 1 cm end has 18/9 ball joint.

C — Five percent platinum asbestos plug, loose packed acting as catalyst to convert SO_2 to SO_3, optimum temperature for SO_2 conversion is about 400°C.

D — Heavy duty combustion assembly, consisting of two 20-cm long independently controlled furnaces. One furnace is stationary, and the other is movable. Maximum temperature attainable is 1 000°C.

E — Combustion boat about 10 cm in length.

6, 7, 8 — Scrubbers for removal of sulphur compounds and water formed by combustion; each bottle is of 250 ml capacity. Glass wool plugs are provided to absorb solutions, which may foam over as they become exhausted.
No. 6 contains chromic acid (375 g CrO_3 + 750 ml of water).
No. 7 contains concentrated sulphuric acid saturated with chromium oxide (900 ml H_2SO_4 + 50 g CrO_3).
No. 8 contains concentrated sulphuric acid (98 percent) for moisture removal. It can be eliminated, since its only purpose is to prolong the life of phosphorus pentoxide and glass wool in tube No. 10.

9 — Special ground glass connection (10 mm tubing 12 cm long blown on 18/9 socket joint. Glass wool plug is an added guard against foaming and mist formation in the preceding scrubbers.

13 — Safety trap.

11 — Absorption tube: Liquid 30 percent potassium hydroxide

Interior { Glass wool
Activated alumina
Glass wool
Ascarite
Glass wool

FIG. 1 APPARATUS FOR DETERMINATION OF CARBON IN SULPHUR

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Reconnect the oxygen to the combustion tube and, while flow continues, weigh accurately about 2·0 to 2·5 g of the undried sample.

3.7.2.4 For a freshly prepared absorption tube, allow 30 minutes before making the first weighing. To remove the absorption tube, close the valve on the phosphorus pentoxide tube and also the one on the absorption tube itself and remove it from the train. Release the vacuum on the absorption tube by opening the stopper and allowing the solution in the arm to rise to the height of that in the body of the absorption tube. Close the absorption tube. Record the weight of the tube. Replace the tube in the train, observing the following precautions:

> Open the valve on the phosphorus pentoxide tube for 5 seconds then close the valve before attaching the absorption tube. Attach the tube and open its valve. Then open valve on phosphorus pentoxide tube.

3.7.2.5 Repeat the step of **3.7.2.4** every 15 minutes until two successive weighings agree within 0·5 mg.

3.7.2.6 Open the combustion tube and place the boat containing the sample about 1·5 cm from the front edge of the furnace. Close the combustion tube. Increase the oxygen feed by carefully opening the oxygen feeder valve. Ignite the sample by heating the combustion tube with a gas burner at the front end of the boat until a flame is noted. Immediately move the burner about 5-8 cm ahead of the boat, maintaining just enough heat to keep the sulphur burning.

At the completion of burning, the carbon flake will burn with a bright flame, leaving only a carbonaceous stain on the boat. This stain is burnt by moving the furnace over the boat. Allow the furnace to remain over the boat for about five minutes and then return it to its original position. Allow thirty minutes for flushing all of the carbon dioxide and then remove the absorption tube and calculate carbon. Carry out a blank experiment in a similar manner without the sample.

3.7.3 *Calculation*

$$\text{Carbon, percent by mass} = \frac{(M_1 - M_2)}{M\left(1 - \frac{P}{100}\right)} \times 27{\cdot}29$$

where

M_1 = mass in g gained from the sample,

M_2 = mass in g gained from the blank experiment,

M = mass in g of the sample taken for the test, and

P = percentage moisture as determined in **3.2**.

3.8 Determination of Fineness

3.8.1 *Procedure* — Weigh accurately about 30 g of the sample and transfer to an appropriate standard sieve provided with a lid. Shake the sieve till only coarse particles remain on the sieve. Break up any soft agglomerates by gentle pressure and again shake till all fine sulphur has passed. Weigh the residue of coarse particles and calculate the percentage of the material retained on the specified sieve.

3.9 Determination of Oil and Bituminous Matter

3.9.1 *Apparatus*

3.9.1.1 *Volumetric flask* — 100 ml capacity.

3.9.1.2 *Soxhlet flasks* — 250 ml capacity.

3.9.1.3 *Wiley continuous extractor* (*without crucible or thimble*) — Roll up a sheet of copper foil 10 cm × 40 cm in such a way that a spiral is formed without contact between the surfaces. Leave a tab about 10 mm square on one corner and punch a hole in this tab so that the coil may be lifted from the Wiley tube with a wire hook. Keep this tab in the centre of the coil.

3.9.2 *Reagents*

3.9.2.1 *Petroleum ether* — redistilled, boiling range 40 to 60°C.

3.9.2.2 *Dilute nitric acid* — about 5 N.

3.9.3 *Procedure*

3.9.3.1 Weigh into the 100-ml flask 50 g of the powdered sample, or a smaller quantity, if the oil content is known to be high. Add about 50 ml of petroleum ether, shake the contents thoroughly every half an hour for about four hours and allow to settle. Decant the solution through a filter into the Wiley extraction tube, shake the residual sulphur a second time with about 30 ml of the solvent. Allow it to settle and filter as before. Continue washing and shaking the sulphur with fresh portions of the ether until about 175 ml of the solvent have been used or until there is sufficient to fill the tube above the copper foil.

3.9.3.2 Clean the copper coil with dilute nitric acid, wash with water, then with alcohol, dry with ether and place in Wiley tube. Then boil the petroleum ether in the tube until all the sulphur has been deposited on the copper as copper sulphide (CuS). Transfer the solvent to the weighed flask filtering, if necessary, and wash the coil and the tube, and filter with small quantities of petroleum ether. Evaporate the solvent on a steam-bath, dry to constant mass and weigh. Carry out a blank simultaneously. Calculate the percentage of oil and bituminous matter.'

INTERNATIONAL SYSTEM OF UNITS (SI UNITS)

Base Units

Quantity	*Unit*	*Symbol*
Length	metre	m
Mass	kilogram	kg
Time	second	s
Electric current	ampere	A
Thermodynamic temperature	kelvin	K
Luminous intensity	candela	cd
Amount of substance	mole	mol

Supplementary Units

Quantity	*Unit*	*Symbol*
Plane angle	radian	rad
Solid angle	steradian	sr

Derived Units

Quantity	*Unit*	*Symbol*	*Conversion*
Force	newton	N	1 N = 0·101 972 kgf
Energy	joule	J	1 J = 1 N.m
Power	watt	W	1 W = 1 J/s
Flux	weber	Wb	1 Wb = 1 V.s
Flux density	tesla	T	1 T = 1 Wb/m^2
Frequency	hertz	Hz	1 Hz = 1 c/s (s^{-1})
Electric conductance	siemens	S	1 S = 1 A/V
Pressure, stress	pascal	Pa	1 Pa = 1 N/m^2

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